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(54) Abstract Title

Iodine-releasing disinfectant solution

(57) An aqueous iodine-releasing disinfectant solution comprises an iodophor, a disinfectant active organic acid and an anionic alkyl diphenyl ether disulphonate surfactant. The preferred composition is a concentrated solution which, on dilution with water, yields 15-30 ppm of available iodine, 300-700 ppm of acid and 300-700 ppm of surfactant. Alternatively, the composition can be in ready-to-use form with the active ingredients present in the aforementioned amounts.

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IMPROVEMENTS IN ORGANIC CHEMICAL COMPOSITIONS

This invention relates to disinfectant iodine-releasing cleaning compositions for hard surfaces. More particularly, it relates to such compositions in which the amount of iodine has been reduced to a level at which the iodine does not present a staining problem, and yet the disinfectant properties of the compositions remain as high as those with considerably larger amounts of available iodine.

Iodine was discovered in the early years of the 19th century and has long been used for a wide variety of germicidal and disinfecting purposes for both medical and non-medical uses. Iodine is an effective antiseptic but has certain drawbacks, such as an unpleasant smell and the tendency to produce dark coloured stains on surfaces. Furthermore, it is only sparingly soluble.

In order to overcome the drawbacks associated with the use of iodine, the iodophors were developed. Iodophors are generally defined as complexes of iodine and surface active agents, particularly nonionic detergents, which act as carriers and solubilizing agents for the iodine. Iodophors generally enhance the bactericidal activity of iodine, reduce its odour and reduce its staining characteristics. Typical surfactants used in the preparation of iodophors are polyvinyl pyrrolidone (povidone), polyether glycols, polyvinyl alcohols, polyacrylic acids, polyamides, polyoxyalkylenes and the like. The most commonly used surface active agents include polyvinyl pyrrolidone and its iodine complex generally referred to as "povidone-iodine". Other commonly used surface active agents in iodophors include long chain alkyl phenol/ethylene oxide

condensates. Although nonionic, anionic and cationic surface active agents can be used as complexing agents for iodine, nonionic surfactants are preferred and most of the commercially available iodophors are nonionic in nature. There is an extensive amount of patent literature which discusses iodophors; and more details on the chemistry of iodophors can be found in W. Gottardi, "Iodine and Iodine Compounds", which is chapter 8 of Block, Disinfection, Sterilization and Preservation (1983), pertinent portions of which are incorporated herein by reference.

The use of iodophors in disinfectant compositions for hard surface cleaning is well known in the art, as exemplified in Bakka U.S. Patent No. 3,650,966, Morlock et al. U.S. Patent No. 4,088,597, Langford U.S. Patent No. 4,207,310, and Gartner U.S. Patent No. 4,526,751. It is generally believed that, in order to provide effective "hospital strength" disinfection, iodophor-containing cleaning/disinfectant compositions must provide at least 75 parts per million (ppm) of available iodine. However, the use of iodine at this level produces dark stains, particularly on porous surfaces. Of course, reducing the level of available iodine will reduce staining but iodine levels below 75 ppm do not generally produce acceptable disinfection.

Kolstad U.S. Patent No. 4,867,897 discloses a germicidal composition comprising iodine, a solvent for the iodine, and one or more anionic detergents. The preferred anionic detergents are sodium lauryl sulphate and nonylphenoxytetra(ethyleneoxy)ethanol sulphates. The stated purpose of these compositions is to provide a level of available iodine which is higher than what would ordinarily be attainable with the use of iodophors. It is readily apparent that such compositions, because of

their high iodine level, would be likely to cause severe staining on porous surfaces.

5 There is therefore a need to provide an iodine releasing detergent/disinfecting composition in which iodine levels can be reduced to the point where there is no significant staining of porous surfaces whilst, at the same time, providing an iodine-releasing composition which exhibits hospital strength disinfection.

10 The invention significantly reduces the staining of surfaces by iodine-based disinfectant compositions. The compositions of this invention comprise a nonionic iodophor, an organic acid such as citric acid, and an anionic surfactant which is an alkyl phenoxybenzene disulphonate. The presence of the organic acid and this
15 type of anionic surfactant permits a significant reduction in the amount of iodophor, whilst still maintaining hospital strength disinfectant activity. The reduction in the amount of the iodophor, and hence in the amount of available iodine, results in reduced staining
20 whilst still maintaining a level of disinfection achieved with higher levels of iodine. Typically, iodine levels can be reduced from the 75 ppm thought to be required for hospital strength disinfection to a level of about 25 ppm or even lower in some circumstances.

25 More particularly, this invention provides a concentrated aqueous iodine releasing disinfectant solution comprising (a) an iodophor, (b) a disinfectant active organic acid in sufficient amount to provide from about 300 to about 700 ppm of said acid, and (c) an
30 anionic alkyl diphenyl ether disulphonate surfactant in sufficient amount to provide from about 300 to about 700 ppm of said surfactant; this solution yields, on dilution

with water, from about 15 to about 30 ppm of available iodine.

The invention also provides an aqueous iodine-releasing disinfectant composition which comprises (a) a nonionic iodophor in sufficient amount to provide the composition with from about 15 to about 30 ppm of available iodine, (b) 300 to 700 ppm of a disinfectant active organic acid, and (c) 300 to 700 ppm of an anionic alkyl diphenyl ether disulphonate surfactant.

The iodophors preferred for use in the present invention are complexes of iodine with nonionic surface active agents such as povidone, alkylphenol ethoxylates, secondary alcohol ethoxylates, ethylene oxide/propylene oxide block copolymers and butoxy-ethylene oxide/-propylene oxide block copolymers. A preferred commercially available iodophor is α -(p-nonylphenyl)- ω -hydroxypoly(oxyethylene)-iodine complex sold under the trademark Clean Front Concentrate by West Agro which provides 20% titratable iodine. Other commercially available iodophors include Biopal NR-20 from Rhône Poulenc, a nonylphenoxy poly (ethyleneoxy) ethanol-iodine complex having 20-24% titratable iodine and Bio-Surf I-20, an iodine complex of similar chemical structure from Lonza Inc. Also available from West Agro are MS/DS Concentrate, a polyethoxy/polypropoxy/ polyethoxyethanol-iodine complex having 12.9% titratable iodine, and Lo Foam Concentrate, a mixture of butoxy polypropoxy/polyethoxyethanol-iodine complex and polyethoxy/polypropoxy/polyethoxyethanol-iodine complex having 12% titratable iodine.

The term "titratable iodine", sometimes referred to as "available iodine", is determined by titration with a standard thiosulphate solution and microbial activity is related directly to this value. In iodine complexes,

the available iodine usually amounts to from about 80% to about 85% of the iodine originally added.

5 The purpose of the acid is to promote the germicidal activity of the iodophor in order to provide additional sanitizing effect and also to provide a pH in the acid range. Any suitable acid can be used for this purpose; these include hydrochloric acid, lactic acid, hydroxyacetic acid, phosphoric acid, acetic acid, boric acid, citric acid, oxalic acid and the like. Organic acids are preferred and, of these, citric acid is particularly preferred. A pH in the acid range functions to maintain the availability of iodine in the solution. Generally, the pH of the solution should be below 6 and preferably between 2.0 and 5.0.

15 Although anionic detergents in general have been previously known as possible ingredients in iodine-releasing disinfectant solutions, it has now been discovered that the use of anionic detergents which have two aromatic rings, particularly phenyl rings, provides a level of effectiveness not attained by other anionic surfactants. Preferred anionic surfactants for use in connection with this invention include the disulphonates such as Dowfax 2A1 (or 2EP or XDS 30599 or XU 40333.00) sold by the Dow Chemical Corporation and Poly-Tergent 2A1 (or 2EP) sold by Olin Corporation, both of which comprise principally sodium dodecyldiphenyl ether disulphonate. Other suitable surfactants include: disodium mono- and di-dodecyl diphenyl oxide disulphonate (Aerosol DPOS-45, American Cyanamid); sodium alkyl diphenyl oxide disulphonate (Calfax DB-45, 10L-45 and 16L-35, Pilot Chemicals Co.); sodium hexyl diphenyloxide disulphonate (Dowfax XDS 8292.00, Dow Chemical Corp.); sodium n-decyl diphenyloxide disulphonate (Dowfax 3B2 or 8390 or XU40340.00, Dow Chemical Corp.); sodium n-hexadecyl diphenyloxide disulphonate (Dowfax XU 40341.00, Dow

Chemical Corp.); n-decyl diphenyloxide disulphonate (Dowfax 3B0, Dow Chemical Corp.); sodium alkyl diphenyl oxide disulphonate (Larosol NRL-40, PPG Industries); sodium decyl diphenyl ether disulphonate (Poly-Tergent 3B2, Olin Corp.); alkyl diphenyl ether disulphonate sodium salt (Poly-Tergent 4C3, Olin Corp.); and sodium dodecyl diphenyloxide disulphonate (Sandoz Sulfonate 2A1, Sandoz Chemicals Corp.).

In addition to the foregoing three ingredients, the compositions of the present invention may also include one or more nonionic surfactants to help solubilize the iodine. Suitable surfactants include fatty alcohol alkoxylates, for example, ethoxylated and propoxylated linear fatty alcohols such as Olin Corp.'s SL series surfactants and Plurafac-D25 by BASF, linear primary alcohol ethoxylates such as Shell Chemical's Neodols, secondary alcohol ethoxylates such as Union Carbide's Tergitol 15-S series, ethylene oxide/propylene oxide block copolymers such as BASF's Pluronic and Tetronics, and alkylphenol ethoxylates such as Union Carbide's Tergitol NP series and Triton X and N series.

The compositions of this invention include disinfectant/detergent compositions which comprise a sufficient amount of an iodophor to provide, under conditions of use, from about 15 to about 30 ppm of available iodine. By "available iodine" is meant the amount of I_2 which is available to function as a disinfectant agent. This amount will, of course, vary according to the particular iodophor used in the composition, and also according to the amounts of the other ingredients present in the composition. The appropriate level of iodophor to use can be easily determined by persons skilled in the art. It has been found that, in order to produce hospital strength disinfection comparable to what would ordinarily be

provided by an aqueous solution of an iodophor -- i.e., about 75 ppm -- this invention requires that the iodophor be present in sufficient amount to yield only approximately 25 ppm of available iodine. If disinfection at a level somewhat lower than hospital strength is all that is necessary for the desired use, an iodine availability level of 15 ppm can be sufficient.

In ready-to-use disinfectant/detergent compositions of this invention, the amounts of organic acid and anionic detergent should desirably be from about 300 to about 700 ppm, preferably from 400 to 600 ppm. With a iodine availability of 25 ppm, suitable levels of organic acid and anionic detergent are each about 500 ppm.

Although the invention includes ready-to-use aqueous compositions of the type described above, it will in most cases be more convenient to supply the disinfectant/ detergent composition in concentrated form, for example in a concentration which, on 1:100 dilution in water, would yield the above-described ready-to-use compositions. Alternatively, where a user is likely to be satisfied with a lower level of disinfection, concentrated solutions can be provided with directions for use which would yield different levels of disinfective activity.

A further reason for formulating the compositions of this invention as dilutable concentrates is that, in concentrated form, the iodine is more stable.

This invention will be better understood by reference to the following examples, which are included here for purposes of illustration and are not intended to be limitations.

Example 1

Procedure for Potentiometric Titration of Available Iodine

This method is intended to give an accurate titration by using a potentiometer and graphing the millivolt readings to obtain a potential curve. The curve in turn is used to find the end point of the titration and the milliliters of titrant.

Equipment: Beckman zeromatic pH meter or equivalent, burette and magnetic stirrer.

Reagents: 0.025 N $\text{Na}_2\text{S}_2\text{O}_3$, 99% Acetic Acid, 2% starch indicator solution (potato starch) and KI crystals.

Procedure: Set up of Beckman meter - remove reference electrode and plug in platinum electrode. Press button marked \pm 700 millivolts. Press read button when you have the electrodes in the sample.

Weigh approximately 0.3 grams of concentrated Iodoform or equivalent amount in formulation to the nearest milligram. Dissolve the sample in 200 ml of distilled water, put on stirrer and immerse the electrodes, then press READ button on pH meter. Add 1 ml of acetic acid, 1 ml of starch indicator and sprinkle KI crystals until the millivolt reading is approximately - 80mv. Begin titrating and plot the points on linear graph paper. Continue till there is a sharp change in potential, then do two more points to be sure this is the end point. For very low concentrations, use 0.001 N $\text{Na}_2\text{S}_2\text{O}_3$.

Calculations

$$\% \text{ available } \text{I}_2 = \frac{\text{ml of titrant} \times \text{N of titrant} \times 12.692}{\text{wt. of sample}}$$

Example 2

A concentrated iodophor-containing detergent/disinfectant composition was prepared having the

following ingredients, in their indicated percentages by weight

Table I

Poly-Tergent SL-62 ⁽¹⁾	3.00
Poly-Tergent 2A1 (45%) ⁽²⁾	11.12
Clean Front Concentrate ⁽³⁾	1.25
Deionized Water	79.63
Citric Acid	5.00

This formulation was diluted 1 part to 100 with tap water and tested against Staphylococcus aureus, Salmonella choleraesuis and Pseudomonas aeruginosa in the AOAC use dilution test specified by the United States Environmental Protection Agency. In this test, the bacteria in question are dried on a surface, then subjected to the test liquid at the specified dilution. The bacteria are then incubated in a nutrient bath. To pass the test, only one in 60 samples of bacteria is allowed to grow when put into the nutrient broth. If the formula is effective against all three organisms, it is considered a "hospital strength disinfectant". The formulation was tested twice and the results are as follows:

Table II

	<u>Staph.</u>	<u>Salmonella</u>	<u>Pseud.</u>
Test #1	1/60	0/60	0/60
Test #2	1/60	1/60	0/60

¹ Poly(oxyethylene/oxypropylene) - (monoethyl/monooctyl/monodecyl) ether; nonionic detergent, Olin Corporation

² Sodium dodecyldiphenyloxide disulphonate; anionic detergent, Olin Corporation

³ α -(p-nonylphenyl)- ω -hydroxypoly(oxyethylene)-iodine, West Agro

These results indicate that this formula provides hospital strength disinfection with only 25 ppm available iodine instead of the recommended 75 ppm iodine. The iodine availability was measured according to the method of Example 1.

Example 3

A formulation similar to the composition of Example 2 was prepared, but with sodium dodecylbenzene sulfonate (Bio-Soft D-40, Stepan Co.) used as the anionic surfactant. Tests were run against Staphylococcus aureus and Pseudomonas aeruginosa. The results are shown in the following Table III.

Table III

<u>Staph.</u>	<u>Pseud.</u>
3/60	8/60

These data show that the formulation is essentially ineffective against the species tested and cannot be regarded as providing hospital strength disinfection.

Example 4

A formulation similar to the composition of Example 2 was prepared except that the iodine level was adjusted downward to 0.72% at 1:100 dilution. The tested solution had only 15 ppm of available iodine as measured by the method of Example 1. Testing, again done in accordance with the method of Example 2, showed effectiveness against the Staphylococcus and Salmonella species but unsatisfactory results against the Pseudomonas species -- Test #1. In a subsequent test -- Test #2 -- the dilution level was adjusted to 1:64, thereby increasing the level of available iodine to 25 ppm, and the composition was tested against the

Pseudomonas species. In this case, the result was successful. The data are shown in the attached Table IV:

Table IV

	<u>Staph.</u>	<u>Salmonella</u>	<u>Pseud.</u>
5 Test #1	1/60	0/60	4/60
Test #2			1/60

Claims:

1. A concentrated aqueous iodine-releasing disinfectant solution which, on dilution with water, yields from 15 to 30 ppm of available iodine, said solution comprising
 - (a) an iodophor,
 - (b) a disinfectant active organic acid in sufficient amount to provide from 300 to 700 ppm of said acid, and
 - (c) an anionic alkyl diphenyl ether disulphonate surfactant in sufficient amount to provide from 300 to 700 ppm of said surfactant.
2. A disinfectant solution according to Claim 1 which, on dilution with water, yields from 15 to 25 ppm of available iodine, in which the organic acid is present in sufficient amount to provide from 400 to 600 ppm of said acid, and in which the surfactant is present in sufficient amount to provide from 400 to 600 ppm of said surfactant.
3. A disinfectant solution according to Claims 1 or 2 in which, on dilution with water, the available iodine is about 25 ppm, the organic acid is present in sufficient amount to provide about 500 ppm of said acid, and the surfactant is present in sufficient amount to provide about 500 ppm of said surfactant.
4. A disinfectant solution according to any of claims 1-3 in which the acid is citric acid.
5. A disinfectant solution according to any of claims 1-4 in which the surfactant is an alkyl diphenyloxide disulphonate.

6. A disinfectant solution according to Claim 5 in which the surfactant is sodium dodecyldiphenyloxide disulphonate.
7. A disinfectant solution according to any of claims 1-6 in which the pH is below 6.0.
8. A disinfectant solution according to Claim 7 in which the pH ranges from 2.0 to 5.0.
9. An aqueous iodine-releasing disinfectant composition which comprises
 - (a) a nonionic iodophor in sufficient amount to provide the composition with from 15 to 30 ppm of available iodine,
 - (b) 300 to 700 ppm of a disinfectant active organic acid, and
 - (c) 300 to 700 ppm of an anionic diphenyl disulphonate surfactant.
10. A composition according to Claim 9 in which the acid is citric acid.
11. A composition according to Claims 8 or 9 in which the surfactant is an alkyldiphenyloxide disulphonate.
12. A composition according to Claim 11 in which the surfactant is sodium dodecyldiphenyloxide disulphonate.



Application No: GB 9828519.0
Claims searched: 1-12

Examiner: Peter Davey
Date of search: 3 March 1999

Patents Act 1977
Search Report under Section 17

Databases searched:

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK CI (Ed.Q): A5E (EBB, ED, EF, ET)

Int CI (Ed.6): A01N 25/00 25/30 59/00 59/12

Other: Online: WPI, CAS ONLINE

Documents considered to be relevant:

Category	Identity of document and relevant passage	Relevant to claims
A	US 4867897 (MDT), see eg. claims 1 and 5	1 and 9 at least

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.

